

PATENT ABSTRACTS OF JAPAN

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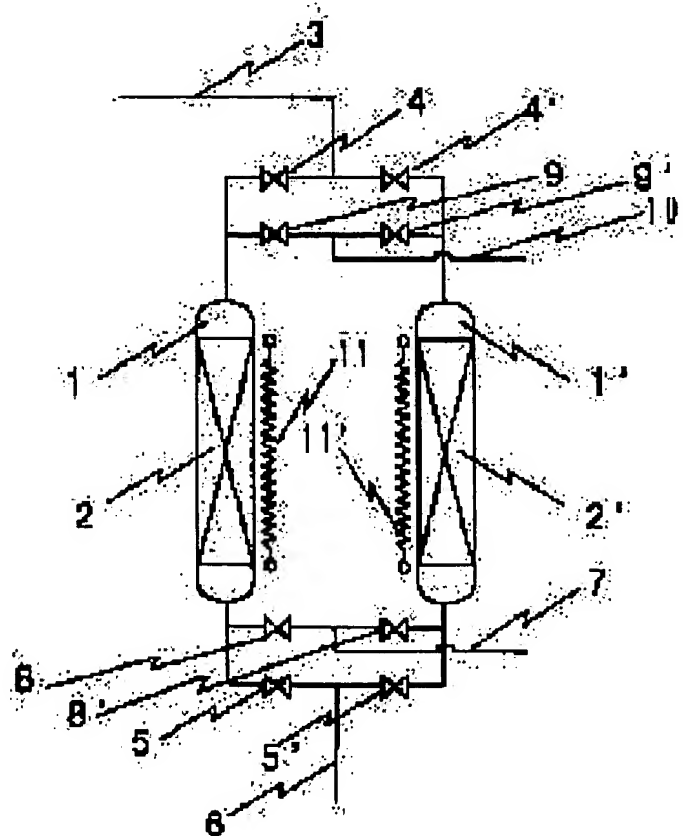
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(54) TREATMENT OF FLUOROCARBON BY DECOMPOSITION AND DECOMPOSITION TREATMENT DEVICE

(57)Abstract:

PROBLEM TO BE SOLVED: To make it possible to continuously decompose a fluorocarbon at a comparatively lower temperature and with high efficiency by bringing a gas containing the fluorocarbon into contact with an eliminator containing an aluminum oxide as an active principle at a specific temperature to decompose the fluorocarbon and thereby forming aluminum fluoride.

SOLUTION: When a gas containing a fluorocarbon is treated with a fluorocarbon decomposition treatment device structured of elimination columns 1, 1' aligned in two parallel rows, eliminators 2, 2' containing an aluminum oxide as an active principle are charged in the elimination columns 1, 1', respectively. The fluorocarbon is decomposed by bringing the gas into contact with the eliminators 2, 2' at 400°C or higher to decompose the fluorocarbon and, consequently, aluminum



fluoride is formed. Further, the eliminators 2, 2' which are no longer capable of decomposing the fluorocarbon are regenerated by bringing the eliminators 2, 2' into contact with the gas containing water vapor under heat. The fluorine fixed as the aluminum fluoride in the eliminators 2, 2' is discharged as hydrogen fluoride, and aluminum is recycled as aluminum oxide.

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CLAIMS

[Claim(s)]

[Claim 1] A decomposition treatment method of fluorocarbon contacting gas containing fluorocarbon to a damage elimination agent which makes an aluminum oxide an active principle at temperature of not less than 400 **, decomposing this fluorocarbon, and making aluminum fluoride form.

[Claim 2] A decomposition treatment method of fluorocarbon contacting gas containing fluorocarbon to a damage elimination agent which makes an aluminum oxide an active principle under coexistence of a steam and/or oxygen at temperature of not less than 400 **, decomposing this fluorocarbon, and making aluminum fluoride form.

[Claim 3] A decomposition treatment method of fluorocarbon reproducing and carrying out the reuse of the damage elimination agent after carrying out decomposition treatment of the fluorocarbon with claim 1 or the decomposition treatment method according to claim 2 by heating under coexistence of a steam at temperature of not less than 400 **.

[Claim 4] A damage elimination pipe filled up with a damage elimination agent which makes an aluminum oxide an active principle is connected to two or more pipes parallel, A decomposition treatment method of fluorocarbon performing decomposition treatment of fluorocarbon continuously by decomposing fluorocarbon by at least 1 pipe, and reproducing a damage elimination agent by at least 1 pipe by changing a gas passageway in the meantime.

[Claim 5] Fluorocarbon CH_3F , CH_2F_2 , CHF_3 , $\text{C}_2\text{H}_2\text{F}_4$, and $\text{C}_2\text{H}_4\text{F}_2$, A decomposition treatment method of the fluorocarbon according to any one of claims 1 to 4 which is at least one sort chosen from CF_4 , C_2F_6 , C_3F_8 , C_4F_8 , and C_5F_{12} .

[Claim 6] Gas which contains fluorocarbon in a damage elimination pipe with which a damage elimination agent which makes an aluminum oxide an active principle was filled up Independence, Or make it circulate under a steam and/or oxygen coexistence, and this damage elimination agent is made to contact, Decomposition treatment equipment of fluorocarbon having reproduced and considering a damage elimination agent after carrying out decomposition treatment by changing fluorine in this fluorocarbon into aluminum fluoride as composition which can carry out a reuse by heating under coexistence of a steam.

[Claim 7] A pipe with which a damage elimination agent which makes an aluminum oxide an active principle was filled up is connected to two or more pipes parallel, Decomposition

treatment equipment of fluorocarbon having composition which can perform decomposition treatment of fluorocarbon continuously by decomposing fluorocarbon by at least 1 pipe, and reproducing a damage elimination agent by at least 1 pipe by changing a gas passageway in the meantime.

[Claim 8] Fluorocarbon CH_3F , CH_2F_2 , CHF_3 , $\text{C}_2\text{H}_2\text{F}_4$, and $\text{C}_2\text{H}_4\text{F}_2$, Decomposition treatment equipment of the fluorocarbon according to claim 6 or 7 which is at least one sort chosen from CF_4 , C_2F_6 , C_3F_8 , C_4F_8 , and C_5F_{12} .

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention relates to the decomposition treatment method and decomposition treatment equipment of fluorocarbon content gas. More particularly, it is used for a refrigerant, a foaming agent, a solvent, etc., and also it is used by a semiconductor manufacturing process etc., or is related with the decomposition treatment method and decomposition treatment equipment of hydronaliumfluorocarbon and perfluorocarbon which carry out a byproduction by the scouring process of aluminum.

[0002]

[Description of the Prior Art]It is widely used as a refrigerant, a foaming agent, a solvent, etc., and also fluorocarbon, such as hydronaliumfluorocarbon by which a part of hydrogen of hydrocarbon was replaced by fluorine, and perfluorocarbon in which all the hydrogen was replaced by fluorine, is used in the semiconductor manufacturing process etc.

[0003]Also among fluorocarbon, CF_4 and C_2F_6 , The amount used is increasing with

development of semiconductor industry, such as being used as cleaning and the etching agent of a silicon wafer surface, the cleaning agent of a plasma CVD chamber, and the doping substance for optical fibers and the etching agent at the time of liquid crystal TFT manufacture. However, this gas is small, and at a room temperature, such as hardly reacting also to acid or alkali, the solubility to water is dramatically stable, and also is stable under an elevated temperature. To discharge in the used post-open air from it being a substance which brings about global warming, it is necessary to carry out decomposition treatment of these fluorocarbon in advance of discharge. In the scouring process of aluminum, since generation discharge of the CF_4 is carried out in large quantities, it is necessary to carry out decomposition treatment also of this CF_4 .

[0004]It is necessary to carry out decomposition treatment of the chlorofluorocarbon which replaced hydrogen of hydrocarbon with fluorine and chlorine in advance of the used post-discharge from it being a substance which destroys an ozone layer. For this reason, various kinds of researches on the decomposition treatment of chlorofluorocarbon are made until now. As a method of carrying out decomposition treatment of the thing including chlorofluorocarbon

and fluorocarbon generally called chlorofluorocarbon, decomposing methods, such as ** combustion-heat part solution, ** plasma decomposition method, ** supercritical water part solution, ** reagent part solution, and ** catalyst part solution, are known from the former. [0005] However, there were the following problems in these decomposition treatment methods. That is, by ** combustion-heat part solution, if it is not an elevated temperature in CF_4 of what can disassemble common chlorofluorocarbon, sufficient cracking severity will not be acquired, but by the burning conditions into which chlorofluorocarbon may be made to fully disassemble, the inconvenience of being generated in large quantities by nitrogen oxides is during combustion. ** As for a plasma decomposition method, the running cost concerning electric power, plasma gas, etc. is high, and there is a problem in economical efficiency. ** Since a supercritical water part solution needs the high voltage of 320 atmospheres or more at the temperature of 400 **, a processing unit not only becomes large-sized, but it has the inconvenience which becomes big-ticket [a processing cost]. ** Although a reagent part solution is the method of disassembling chlorofluorocarbon reductively using sodium NAFTA RENIDO, there is inconvenience that the stable cracking severity is not acquired. ** catalyst part solution is a hydrolyzing method which used the precious metal catalyst under the elevated temperature.

Throughput sufficient since a catalyst is corroded by hydrogen fluoride which is a decomposition by-product of chlorofluorocarbon is not acquired. In order to solve this problem, research of the ***** object excellent in corrosion resistance has been done, but the still effective thing is not found out.

[0006]

[Problem(s) to be Solved by the Invention] Although various kinds of chlorofluorocarbon decomposing methods are studied as mentioned above, decomposition treatment cannot be carried out, even if it is thermally and chemical very extremely stable in the case of perfluorocarbon, such as CF_4 , and uses the above-mentioned treatment technique for it especially. From these things, development of the method of carrying out decomposition treatment of the fluorocarbon division perfluorocarbon efficiently and continuously at a comparatively low temperature was desired strongly.

[0007]

[Means for Solving the Problem] A result of having repeated research wholeheartedly so that this invention persons may get a method of carrying out decomposition treatment of the fluorocarbon which solves a fault of these conventional technologies and brings about global warming, It found out decomposing fluorocarbon and making aluminum fluoride form by contacting gas containing fluorocarbon at temperature of not less than 400 ** to a damage elimination agent which makes an aluminum oxide an active principle. It found out that decomposition treatment capability of fluorocarbon increased by making a steam live together in gas containing fluorocarbon. What can be reproduced and done for a reuse by heating a damage elimination agent after carrying out decomposition treatment of the fluorocarbon under steam coexistence, And by combining decomposition treatment of fluorocarbon, and reproduction operation of a damage elimination agent, it found out that decomposition treatment of the fluorocarbon could be carried out efficiently continuously etc., and this

invention was completed.

[0008] That is, this invention is a decomposition treatment method of fluorocarbon contacting gas containing fluorocarbon to a damage elimination agent which makes an aluminum oxide an active principle at temperature of not less than 400 **, decomposing this fluorocarbon, and making aluminum fluoride form.

[0009] This invention, It is a decomposition treatment method of fluorocarbon contacting gas containing fluorocarbon to a damage elimination agent which makes an aluminum oxide an active principle under coexistence of a steam and/or oxygen at temperature of not less than 400 **, decomposing this fluorocarbon, and making aluminum fluoride form.

[0010] Furthermore, this invention is also a decomposition treatment method of fluorocarbon which is reproduced and is characterized by carrying out a reuse by heating a damage elimination agent after carrying out decomposition treatment of the fluorocarbon by a damage elimination agent which makes an aluminum oxide an active principle under coexistence of a steam at temperature of not less than 400 **.

[0011] This invention connects to two or more pipes parallel further again a damage elimination pipe filled up with a damage elimination agent which makes an aluminum oxide an active principle, It is a decomposition treatment method of fluorocarbon performing decomposition treatment of fluorocarbon continuously by changing a gas passageway by decomposing fluorocarbon by at least 1 pipe, and reproducing a damage elimination agent by at least 1 pipe in the meantime.

[0012] In addition, a damage elimination pipe with which a damage elimination agent which makes an aluminum oxide an active principle was filled up, this invention. Circulate gas containing fluorocarbon under independence or a steam, and/or oxygen coexistence, and this damage elimination agent is made to contact, It is decomposition treatment equipment of fluorocarbon having reproduced and having composition which can carry out a reuse by changing fluorine in this fluorocarbon into aluminum fluoride by heating a damage elimination agent after carrying out decomposition treatment under coexistence of a steam.

[0013] Furthermore, in addition, a damage elimination pipe with which a damage elimination agent to which this invention makes an aluminum oxide an active principle was filled up is connected to two or more pipes parallel, It is decomposition treatment equipment of fluorocarbon having composition which can perform decomposition treatment of fluorocarbon continuously by changing a gas passageway by decomposing fluorocarbon by at least 1 pipe, and reproducing a damage elimination agent by at least 1 pipe in the meantime.

[0014]

[Embodiment of the Invention] This invention is applied to the decomposition treatment of the gas containing fluorocarbon. As fluorocarbon which is the target of decomposition treatment, it is applied to hydronaliumfluorocarbon and all perfluorocarbon. As hydronaliumfluorocarbon, for example, CH_3F , CH_2F_2 , There are CHF_3 , $\text{C}_2\text{H}_2\text{F}_4$, and $\text{C}_2\text{H}_4\text{F}_2$ etc., As perfluorocarbon, CF_4 , C_2F_6 , C_3F_8 , C_4F_8 , C_5F_{12} , etc. are mentioned. It is applied to decomposition treatment containing these fluorocarbon, such as air, nitrogen, oxygen, hydrogen, and rare gas.

[0015] In this invention, a damage elimination agent contains an aluminum oxide as an active principle, and even if an inertness metallic oxide is substantially contained in a reaction, it can

use everything but an aluminum oxide independent. Since the mold goods of the aluminum oxide are marketed as catalyst support and adsorbent, they can also be used.

[0016]Although there is no restriction in particular in the shape of a damage elimination agent, the granular type thing of a globular shape, cylindrical shape, and an infinite form, etc. are usually used. In addition, what was fabricated by honeycomb shape can be used. The size of a damage elimination agent can be suitably chosen according to the gestalt for which a damage elimination agent is used.

[0017]In this invention, it is used as the fixed bed, and also a damage elimination agent can also be used as a moving bed and a fluid bed. Usually, decomposition treatment of the fluorocarbon is carried out by passing the gas which is filled up with a damage elimination agent in a damage elimination pipe, and contains fluorocarbon in this damage elimination pipe, and making a damage elimination agent contact. Not less than 400 °C of temperature to which fluorocarbon and a damage elimination agent are contacted is 500-800 °C preferably. Temperature is difficult for decomposition of fluorocarbon below 400 °C, on the other hand, above 800 °C, the decomposition treatment capability of fluorocarbon declines in connection with the heat deterioration of a damage elimination agent, and also there is inconvenience as which a heat-resistant high material is required of a damage elimination pipe. Although the pressure at the time of carrying out decomposition treatment of the fluorocarbon is usually performed by ordinary pressure, it can also be performed under decompression or application of pressure.

[0018]Although there is no restriction in particular in the rate of flow of the gas which contains fluorocarbon in this invention, it is so preferred to make the rate of flow small that the concentration of the fluorocarbon generally contained in gas is high. For this reason, although a damage elimination pipe is designed according to the concentration of fluorocarbon, the quantity of processing-object gas, etc., fluorocarbon concentration is as follows 0.1% -- by low concentration, hollow cylinder base line speed (LV) 0.5-50 cm/sec comparatively. It is preferred that fluorocarbon concentration designs at about 0.1 to 1% so that hollow cylinder base line speed (LV) may serve as the range of 10 cm/sec or less at the high concentration with 0.05-20 cm/[sec and] and fluorocarbon concentration above 1% in hollow cylinder base line speed (LV). Therefore, it becomes a general standard in 10 cm/sec or less in the case of comparatively high concentration from a semiconductor manufacturing process like [in the case of being discharged regularly].

[0019]Thus, by contacting the gas containing fluorocarbon at the temperature of not less than 400 °C to the damage elimination agent which makes an aluminum oxide an active principle, the fluoride in fluorocarbon is changed into aluminum fluoride, and carbon is emitted as carbon dioxide.

[0020]When contacting an aluminum oxide to the damage elimination agent made into an active principle depending on the kind of fluorocarbon and carrying out decomposition treatment, carbon may deposit on the damage elimination agent surface, or throughput may fall to it. Carbon monoxide may flow out into damage elimination pipe outlet gas. When processing such fluorocarbon, and carrying out decomposition treatment, by making oxygen live together, these problems can be canceled and decomposition treatment can be carried out efficiently. Here, it can carry out remarkably also by [which add pure oxygen as a method of

making oxygen living together, in the gas containing fluorocarbon] adding air.

[0021]When contacting the gas which contains fluorocarbon in this invention at the temperature of not less than 400 °C to the damage elimination agent which makes an aluminum oxide an active principle and carrying out decomposition treatment, the throughput per damage elimination agent unit weight can be increased by making a steam live together. Throughput increases as the steam partial pressure in gas becomes high in this method. However, 150 or less mmHg is usually 100 or less mmHg preferably as a steam partial pressure in gas from producing the fall of producing decline in cracking severity, or throughput, when a steam partial pressure is too high.

[0022]The method of making a steam living together has the method of adding the method of introducing into the damage elimination pipe heated after contacting the gas which contains fluorocarbon, for example in water at ordinary temperature and including moisture, water, or a steam in the gas which contains fluorocarbon at the entrance of a damage elimination pipe, etc. It can carry out according to the conditions when not making the aforementioned steam usually live together as an operating condition at the time of making a steam live in fluorocarbon together and carrying out decomposition treatment to it.

[0023]In this invention, the damage elimination agent to which decomposition treatment of the gas containing fluorocarbon was carried out, and cleaning capacity fell, or the damage elimination agent which lost damage elimination capability is renewable by making the gas which contains a steam under heating contact. After reproducing, a reuse can be carried out as a damage elimination agent. The fluorine which was being fixed as aluminum fluoride in the damage elimination agent is discharged by this regeneration out of a system as hydrogen fluoride, and aluminum is reproduced as an aluminum oxide.

[0024]It can carry out by circulating air, nitrogen, oxygen, hydrogen, rare gas, etc. which contain a steam independent or a steam in the damage elimination pipe heated by the temperature of not less than 400 °C as a regeneration method. 400-800 °C is usually about 500-800 °C preferably from the cooking temperature in the case of reproduction producing the heat deterioration of a damage elimination agent above 800 °C, and the throughput after reproduction declining. Although there is no restriction in particular in the rate of flow of a steam partial pressure and gas, etc., since reproduction takes a long time when a steam partial pressure is low, usual [5 or more mmHg of / 10 or more mmHg of] is 15 or more mmHg more preferably.

[0025]As total pressure of oxygen, hydrogen, nitrogen, air, and rare gas containing the steam independence in the case of reproduction, or a steam, it may especially be ordinary pressure, decompression, or a pressurization state, and is not limited. Although there is no restriction in particular in the rate of flow of the gas containing the thing steam in reproduction operation, it is [sec] usually about 2-100 cm/sec preferably 0.5-150 cm /at hollow cylinder base line speed (LV).

[0026]Thus, reproduction and since a reuse can be carried out, a damage elimination pipe is arranged for a damage elimination agent to two-row parallel, By damage elimination pipe of one of the two, decomposition treatment of the fluorocarbon can be continuously carried out by performing by turns the method of performing decomposition treatment of fluorocarbon and performing reproduction operation of a damage elimination agent to the meantime by

other damage elimination pipes, via a switching valve. In this disposal method, a damage elimination pipe is used as two pipes, and also in an adult case, change of the amount of raw gas can carry out decomposition treatment continuously efficiently by arranging plural lines, such as three pipes and four pipes, via a switching valve. The example in the case of having arranged the damage elimination pipe to two-row parallel by drawing 1 showed the decomposition treatment equipment of the fluorocarbon in this invention.

[0027]In contacting the damage elimination agent after the decomposition treatment of fluorocarbon with a steam in this invention and reproducing, hydrogen fluoride flows out into the gas of a damage elimination pipe exit. Hydrogen fluoride is a toxic substance, and it precedes discharging and it is necessary to process it. Hydrogen fluoride is easily removable by making the depurator which can purify fluorine system gas at ordinary temperature contact. There are a dry type purification method using the depurator (JP,9-9921,A) etc. which use as the main ingredients the depurator (JP,5-237324,A) which uses a zinc oxide as the main ingredients, and strontium hydroxide as such a depurator, and a publicly known wet purification method. By combining these purification methods with the decomposition treatment method of fluorocarbon, perfect processing without the outflow outside of a fluoride can be performed.

[0028]

[Example]Next, this invention is not limited by these although an example explains this invention concretely.

Example 1 catalyst ***** alumina (the product made from Nissan Girdler, H-218A) is crushed, 24.9mL (60 mm of filling length) restoration of the sifted-out alumina of 12-16 meshes was carried out at the damage elimination pipe made from stainless steel 23.0 mm in inside diameter, and 400 mm in length, and it heated at 700 ** with the electric furnace, circulating nitrogen gas. The nitrogen gas which contains 2000 ppm of CF_4 gas in this is circulated under ordinary pressure by the flow of 500 mL/min (hollow cylinder base line speed $LV = 7 \text{ cm}/(\text{sec})$), Outlet gas was monitored by the gas chromatograph (the Shimadzu Corp. make, GC-4C), and time until it makes into a breakthrough the point that CF_4 concentration in outlet gas amounted to 20 ppm and carries out a breakthrough was measured. Throughput (throughput (L) of pure CF_4 to per [alumina 1L]) was searched for by calculation from the result. The result was shown in Table 1.

[0029]

[Table 1]

Table One (L/L damage elimination agent) Example Time to a breakthrough The amount of damage elimination of CF_4 gas (min)

1 2988 120.3[0030]The nitrogen gas containing example 2 CF_4 was considered as moisture saturation at 20 **, and also the decomposition treatment experiment was conducted like Example 1. A result is shown in Table 2.

[0031]

[Table 2]

Table Two (L/L damage elimination agent) Example Time to a breakthrough The amount of

damage elimination of CF_4 gas (min)

2 8988 361.1[0032]The nitrogen gas containing example 3CF_4 was changed into the air containing CF_4 , and also the decomposition treatment experiment was conducted like Example 1. A result is shown in Table 3.

[0033]

[Table 3]

table 3 example (L/L damage elimination agent) Time to a breakthrough The amount of damage elimination of CF_4 gas (min)

3 3598 144.5[0034]Example Changed 4CF_4 into CH_2F_2 , and nitrogen was changed into air, and also the decomposition treatment experiment was conducted like Example 1. A result is shown in Table 4.

[0035]

[Table 4]

Table Four (L/L damage elimination agent) Example Time to a breakthrough The amount of damage elimination of CH_2F_2 (min)

4 5976 240.7[0036]The damage elimination agent after the decomposition treatment used in example 5 Example 1 was heated at 700°C , and the nitrogen gas which carried out moisture saturation at 20°C under ordinary pressure was circulated for 48 hours by the flow of 500 mL/min (hollow cylinder base line speed $\text{LV} = 5 \text{ cm}/(\text{sec})$). Furthermore, dry nitrogen gas was circulated under 700°C heating ordinary pressure after that for 12 hours, and alumina was reproduced. Next, the nitrogen gas which contains 2000 ppm of CF_4 like Example 1 was circulated under ordinary pressure by the flow of 500 mL/min (hollow cylinder base line speed $\text{LV} = 7 \text{ cm}/(\text{sec})$), and the time to a breakthrough was measured. Thus, the reproduction operation of CF_4 decomposition treatment and a damage elimination agent was repeated several times. These results are shown in Table 5. Before and after CF_4 processing, the energy dispersive X-ray analyzer (EDAX) analyzed the damage elimination agent of the entrance side in a damage elimination pipe, and the content of fluorine was investigated. These results are shown in Table 6.

[0037]

[Table 5]

Table Five (L/L damage elimination agent) Damage elimination agent use count Time to a breakthrough The amount (min) of damage elimination of CF_4 gas

First time 2995 120.3 2 2614 105.4 3 2315 93.0 4 218687.8 5 2137 85.9[0038]

[Table 6]

Table Six Damage elimination agent analytical measurement point Fluorine content in a damage elimination agent After first time CF_4 processing 65% (32% of aluminum content)

After first time reproduction 0.5% (53% of aluminum content)

After 2nd CF_4 $^\circ\text{C}$ After 64% (33% of aluminum content) reproduction [2nd first time] 0.4%

(53% of aluminum content)

After 3rd CF₄ processing 65% (32% of aluminum content)

After 3rd first time reproduction 0.6% (52% of aluminum content)

[0039]On the decomposition treatment conditions of example 6 Example 2, the reproduction operation of the same decomposition treatment as Example 5 and a damage elimination agent was repeated (decomposition treatment of CF₄ was performed in the moisture saturation in 20

**, and also it is the same as Example 5). A result is shown in Tables 7 and 8.

[0040]

[Table 7]

Table Seven (L/L agent) Damage elimination agent use count Time to a breakthrough The amount of damage elimination of CF₄ gas (min)

First time 8990 361.1 2 7870 316.1 3 6753 271.2[0041]

[Table 8]

Table Eight Damage elimination agent analytical measurement point Fluorine content in a damage elimination agent After first time CF₄ processing 65% (32% of aluminum content)

After first time reproduction 0.4% (52% of aluminum content)

After 2nd CF₄ processing 64% (32% of aluminum content)

After 2nd first time reproduction 0.6% (53% of aluminum content)

After 3rd CF₄ processing 65% (32% of aluminum content)

After 3rd first time reproduction 0.5% (52% of aluminum content)

[0042]On condition of example 7 Example 3, decomposition treatment and reproduction of the damage elimination agent were repeated like Example 5 (the gas containing CF₄ was changed into air from nitrogen, and also it is the same as Example 6). A result is shown in Tables 9 and 10.

[0043]

[Table 9]

table 9 damage-elimination agent use count (L/L agent) Time to a breakthrough The amount of damage elimination of CF₄ gas (min)

First time 3595 144.5 2 3150 126.5 3 2870 115.0[0044]

[Table 10]

Table Ten Damage elimination agent analytical measurement point Fluorine content in a damage elimination agent After first time CF₄ processing 65% (32% of aluminum content)

After first time reproduction 0.5% (52% of aluminum content)

After 2nd CF₄ processing 64% (33% of aluminum content)

After 2nd first time reproduction 0.4% (52% of aluminum content)

After 3rd CF₄ ** 65% (32% of aluminum content)

After 3rd first time reproduction 0.6% (53% of aluminum content)

[0045]On condition of example 8 Example 4, it reproduced repeatedly like Example 5

(changed CH₄ into CH₂F₂, and nitrogen was changed into air, and also it is the same as Example 5). A result is shown in Tables 11 and 12.

[0046]

[Table 11]

Table 11 (L/L agent) Damage elimination agent use count Time to a breakthrough The amount of damage elimination of CH₂F₂ gas (min)

First time 5980 240.8 2 5280 212.0 3 4930 198.0[0047]

[Table 12]

Table 12 Damage elimination agent analytical measurement point Fluorine content in a damage elimination agent After first time CF₄ processing 65% (32% of aluminum content)

After first time reproduction 0.6% (52% of aluminum content)

After 2nd CF₄ processing 64% (32% of aluminum content)

After 2nd first time reproduction 0.6% (53% of aluminum content)

After 3rd CF₄ processing 65% (32% of aluminum content)

After 3rd first time reproduction 0.4% (52% of aluminum content)

[0048]Two pipes of things which carried out 83mL (200 mm of filling length) restoration of the same damage elimination agent as example 9 Example 1 at the damage elimination pipe made from stainless steel 23.0 mm in inside diameter and 1000 mm in length were manufactured, and heating maintenance of these was carried out with the electric tubular furnace at 700 **. The nitrogen gas which contains 5000 ppm of CF₄ while circulating nitrogen gas in one pipe was circulated under heating ordinary pressure for 24 hours by the flow of 500 mL/min (hollow cylinder base line speed LV = 7 cm/(sec)). In one of other pipes, the nitrogen gas which carried out moisture saturation at 25 ** was circulated under heating ordinary pressure for 20 hours by the flow of 500 mL/min (hollow cylinder base line speed LV = 7 cm/(sec)). Furthermore under 700 ** heating ordinary pressure, dry nitrogen gas was circulated for 4 hours, and the damage elimination agent was reproduced.

[0049]Decomposition treatment of 3 cycle deed and the CF₄ was continuously carried out for these operations by turns. The depurator which uses a zinc oxide as the main ingredients for the gas and reproduction exhaust gas after those decomposition treatment (86% of a zinc oxide) 5% of potassium oxide and the cleaning cylinder which used alumina 9% as the pipe made from stainless steel 150 mm in inside diameter and 500 mm in length in 5.3L restoration (300 mm of filling length) were circulated, and cleaning cylinder outlet gas was analyzed. CF₄ was analyzed by the gas chromatograph with a thermal conductivity detector (GC-4C, Shimadzu Corp. make), hydrogen fluoride was monitored with the indicator tube for hydrogen fluoride (made by Gas Tech company), and nitrogen oxides were monitored with the indicator tube for nitrogen oxides (made by Gas Tech company). These results are shown in Table 13.

[0050]

[Table 13]

Table 13 Kind of measurement gas Cleaning cylinder in 3 cycles Minimum-limit-of-detection concentration Concentration in outlet gas (ppm)

CF₄N.D. 5 HF (F₂) N.D. 0.25 NO N.D. 1 NO₂N.D. 0.5[0051]Comparative example

Decomposition treatment of CF₄ was performed on the same conditions as Example 1 except having changed 1 - 2 damage-elimination agent into a titania and zirconia. As a result, it was admitted that CF₄ was flowing out of immediately after a decomposition experiment start so much into damage elimination pipe outlet gas. Damage elimination pipe outlet gas was analyzed 1 hour after the experiment start, and the cracking severity of CF₄ was measured.

These results were shown in Table 14 with the case in Example 1.

[0052]

[Table 14]

Table 14 Kind of damage elimination agent CF₄ concentration (ppm) cracking severity of the damage elimination pipe outlet gas of 1 hour after (%)

Example 1 aluminum oxide n.d 100 comparative-example 1 titanium oxide 1760 12

Comparative example 2 zirconium oxide 170015[0053]

[Effect of the Invention]According to the decomposition treatment method of the fluorocarbon of this invention, it can decompose very efficiently and the perfluorocarbon which was conventionally difficult to decompose besides hydronaliumfluorocarbon can be detoxicated now. The damage elimination agent after decomposition treatment could be reproduced efficiently, and since [which is parallel and performs decomposition treatment of fluorocarbon, and reproduction of a damage elimination agent by carrying out plural-lines arrangement of the damage elimination pipe in parallel further] things were carried out, decomposition treatment of the fluorocarbon could be carried out efficiently continuously.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1]It is an example of the decomposition treatment equipment which has arranged the damage elimination pipe in this invention to two-row parallel.

[Description of Notations]

- 1 and 1' damage elimination pipe
- 2 and 2' damage elimination agent
- 3 The gas supply line containing fluorocarbon
- 4 and 4' entrance-side switching valve
- 5 and 5' outlet side switching valve
- 6 Exhaust line
- 7 The supply line of the gas containing a reproduction service-water steam
- The gas entrance switching valve for 8 and 8' reproduction
- The gas outlet switching valve for 9 and 9' reproduction
- 10 Resurgent gas exhaust line
- 11 and 11' heater

[Translation done.]

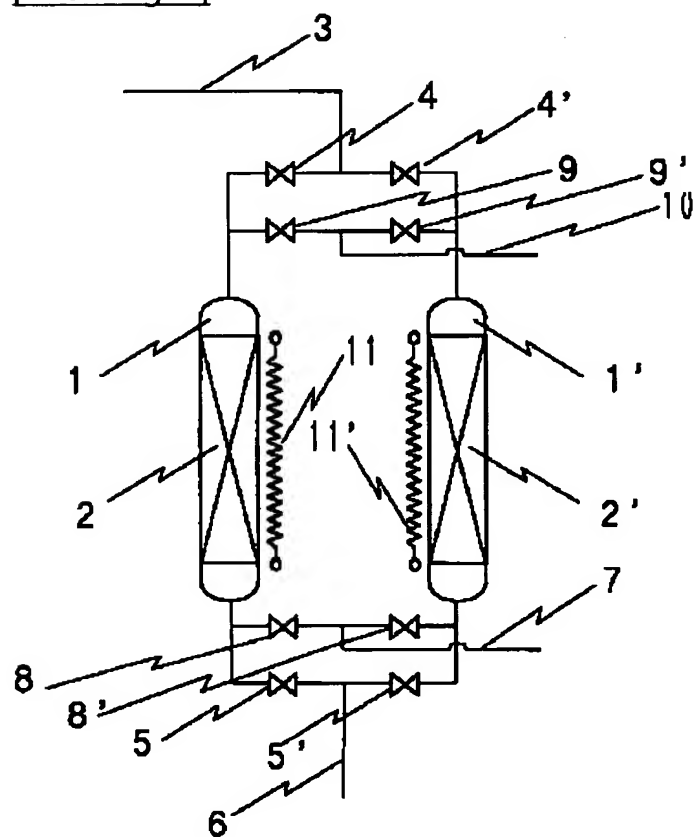
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DRAWINGS

[Drawing 1]



[Translation done.]